

No. 11

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Effect of NUCLEAR RADIATION on Materials at

CRYOGENIC

TEMPERATURES

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JULY 1963-
SEPT. 1963 Report to NASA

LOCKHEED NUCLEAR PRODUCTS

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*** QUARTERLY
PROGRESS
REPORT
No. 11**

, July 1963 through September 1963

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and [1963]*

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**Effect of Nuclear Radiation on materials
at Cryogenic Temperatures ***

PREPARED UNDER

**National Aeronautics/Space Administration
Contract NASw-114**

(NASA

APPROVED BY

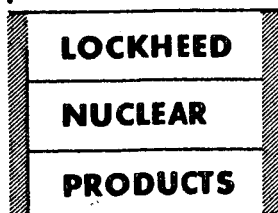
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FOREWORD

This quarterly report is submitted to the Office of Space Launch Vehicles of the National Aeronautics and Space Administration in accordance with the requirements of NASA Contract NASw-114.

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1 INTRODUCTION AND SUMMARY

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This report describes the progress made during the quarter, July through September 1963, on Contract NASw-114.

Preparation for the in-pile screening program continued with calibration of thermocouples on instrumented test specimens against a National Bureau of Standards calibrated platinum resistance thermometer; modification of the test loop carriage assemblies; and check out of the test loop transfer cask and cart.

An addendum to the pedigree report was finalized and published.

Flux measurements were made at various power levels in HB-2 and the results are shown in detail in this report.

AUTHOR

2 EQUIPMENT

2.1 BEAM PORT SHIELD

Flux measurements taken by Lockheed at low reactor power levels in HB-2 verified the fact that the fast neutron flux in this beam port were lower than previously estimated. The fast neutron flux, that is, above 1/2 Mev, was determined to be 2.2×10^4 neutrons per square centimeter, per second, per watt. The previous fast flux estimate in the beam port was greater by a factor of 25 or some 3.0×10^{13} . Since the test specimen in-pile time is a direct function of the neutron flux, the 1/2" thick Mallory 1000 shield plug located on the inside front face of the inner shield assembly was replaced with a 1/2" thick, type 6061-T6 aluminum shield plug as a means of increasing the flux level at the test location. Measurements with both the Mallory 1000 and the aluminum alloy shield plug in place were made.

Calculations made by Lockheed Nuclear Measurements personnel on the assumption of a gamma dose rate incident on the beam port end of 1×10^9 r/hr and an average gamma energy of 1 Mev (energy absorption mass attenuation coefficient of $0.055 \text{ cm}^2/\text{gm}$), show that the gamma dose rate inside the cryostat head would be as follows:

7.7×10^7 r/hr with the 1/2" Mallory 1000 plug

1×10^7 r/hr with the 3/4" Mallory 1000 plug

6×10^6 r/hr with the 1" Mallory 1000 plug

These calculations do not take into consideration any dose rate contributions by tungsten capture gammas.

During high power runs after replacement of the Mallory 1000 shield plug, the aluminum alloy shield plug was found to be unsuitable because nuclear instrumentation installed in the test loops, the fission chamber, and calorimeter became inoperative in the radiation field in the test chamber with this amount of shielding. The temperature difference between the two thermocouples

in the calorimeter was greater than the recorder scale could accommodate. This indicated that the temperature differential was greater than 45°F and the gamma heating rate was greater than the calculated rate on which the design of the calorimeter was based. (Reference Quarterly Progress Report #1, Section 5.11.2; Report #3, Section 12; and Report #7, Section 6.2.) The fission chamber also became inoperative as a result of the high gamma field. (Reference Quarterly Progress Report #1, Section 5.1.1; and Quarterly Progress Report #6, Section 6.1.)

Since the dose rate with the 3/4" Mallory 1000 plug was below 5×10^7 r/hr, which is required for nuclear instrumentation, the 3/4" plug was recommended for reinstallation into the beam port shield. This plug will provide a margin of safety to assure trouble free operation of the nuclear instrumentation.

The aluminum plug was removed without incident, but difficulties were encountered in replacing the 3/4" Mallory 1000 plug. It appeared that the 1/2-20 stainless steel nut in the plug was sliding over the threaded stud on the inner shield with no thread engagement. A diagnosis of this problem indicated that the electroless nickel plating which was on the stud at the time of installation was no longer present. A chemical analysis of the particulate matter from the beam port showed this material to be principally nickel with a small amount of phosphorous. No tungsten was present in this material. This indicates that the material consisted entirely of plating and contained no Mallory 1000. Impressions of the thread form on the stud were taken remotely and the threads were not as deep as the normal thread profile, although they did not appear to be damaged. In an effort to verify the fact that no thread damage had been incurred on the stud, and to reconstruct the cause of this difficulty, bars of Mallory 1000 material were obtained and machined to the configuration specified in the design drawings with the exception that the plating was omitted. Tests were then conducted on these bars to determine the ductility and strength of the threads with respect to the internal thread of an 18-8 stainless steel nut. In all cases, the threads on the stainless steel nuts yielded and the Mallory 1000 threads were undamaged.

All information resulting from the investigation indicated that the major diameter of the stud was originally machined considerably undersize to allow for extensive plating build-up. Thus, the minor diameter of the nut would slip over the stud thread in the "as-machined" but unplated condition. The undersized thread was then built up with nickel plating to conform with the specified design. During prior plug installations and removal, the plating was apparently removed from the threaded portion of the stud allowing the thread configuration to return to the "as-machined" condition. The nut would then slide over the stud without engagement. Since the shield is now radioactive and cannot be removed and handled manually, the repair of the damage must be accomplished by remote techniques.

The present recommended method for repair consists of re-threading the stud with a 7/16-20 thread and leaving the stud unplated. The shield plug would then be modified by the substitution of a stainless steel 7/16-20 lock type nut in place of the stainless steel 1/2-20 nut. A request for approval of this modification has been submitted to NASA and direction to proceed is expected shortly.

2.2 TEST LOOPS

During this reporting period, considerable effort has been expended on the correlation of instrumented test specimens with platinum resistance thermometers especially calibrated by the National Bureau of Standards. This correlation is required to determine the correction factor applicable to the thermocouples which are attached to the instrumented test specimen. The method of constructing this instrumented test specimen is outlined in Quarterly Progress Report #7, Page 15. This correlation was accomplished by packaging the instrumented test specimen and a NBS calibrated platinum resistance thermometer into a single package, thermally insulated from each other by fiberglass and then wrapped in aluminum foil. The package was inserted into modified helium duct assemblies in the test loop head with the thermocouples attached to the specimen in the same manner in

which they will be attached when the test loop is inserted into the reactor. The leads to the platinum resistance thermometer were then run to a Mueller bridge and a galvanometer. The helium refrigerator system was used to lower the test loop temperature to the desired level after which it was allowed to stabilize for approximately 2 hours before any readings were taken. Periodic checks on the platinum resistance thermometer and the test specimen temperature were made to ensure that they were in an equilibrium condition. After reaching an equilibrium condition, several readings were taken at 20-30 minute intervals. After completion of the readings, the refrigerator temperature was changed and data taken at this level. Temperature points below liquid nitrogen level were repeated several times to check for repeatability of data. This procedure was used primarily in the range below liquid nitrogen temperatures because the output of the thermocouples are low and large variations in the data could introduce considerable changes in the indicated temperature.

The calibration data obtained from the three copper-constantan thermocouples on a type 304SS specimen are presented in Figures 1, 2 and 3.

Following these tests with the refrigeration system, a check was made with the thermocouple and platinum resistance thermometer package being inserted into a liquid nitrogen bath. These results are indicated on Figure 4. It may be noted that the slope of the correction factor is essentially a straight line.

During this period, testing of the two polyurethane seals used in sealing the head to the main body of the test loop continued. The present dimensional configuration of the seals appears to be adequate for the intended purposes with the exception of the "V" groove in the periphery, although some difficulty has been encountered in obtaining seals either to the desired dimension or surface finish.

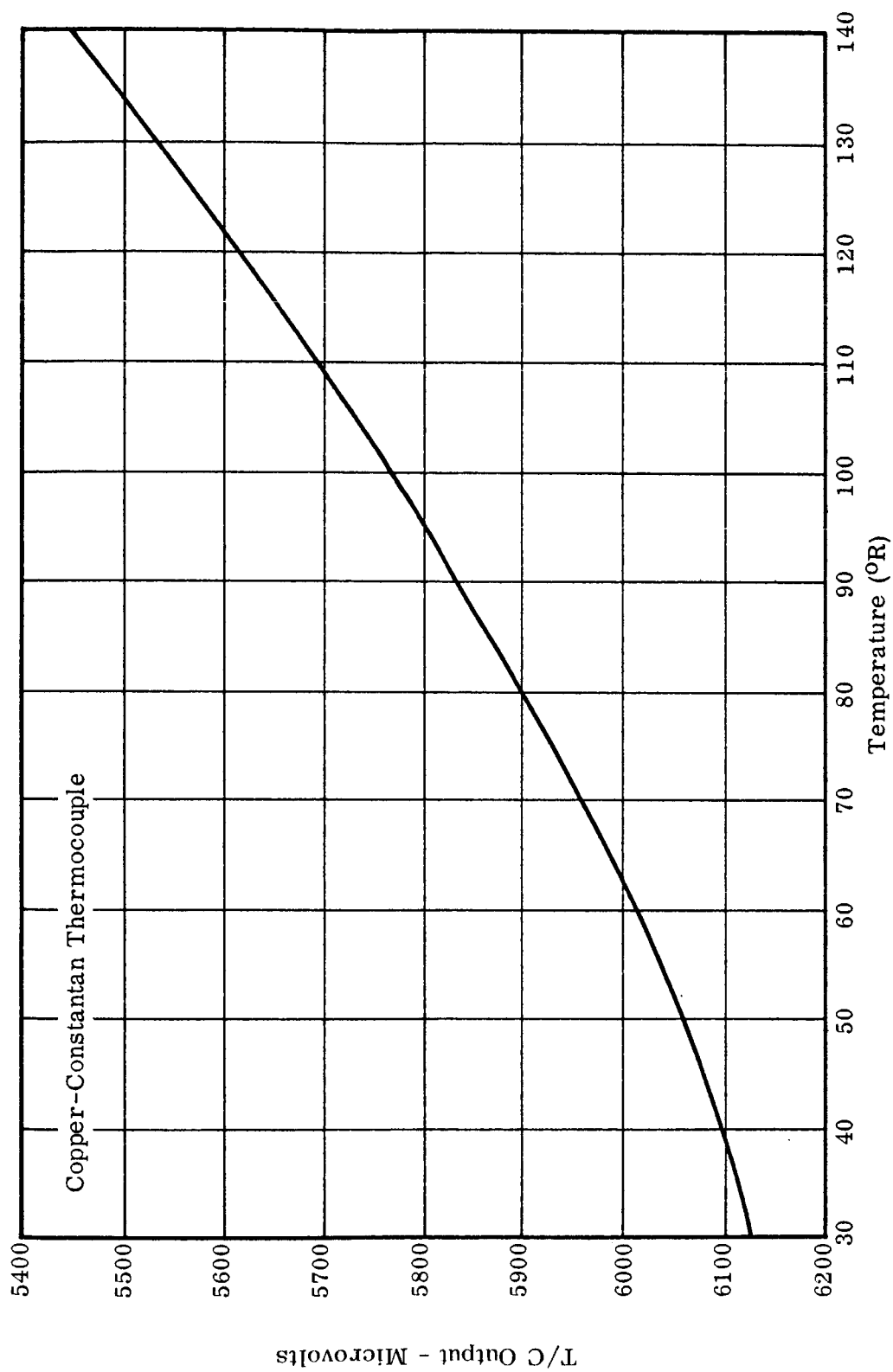


FIGURE 1 304 SST INSTRUMENTED TEST SPECIMEN, PLATINUM RESISTANCE THERMOMETER
COMBINATION PACKAGE IN TEST LOOP #4 - THERMOCOUPLE NO. 1

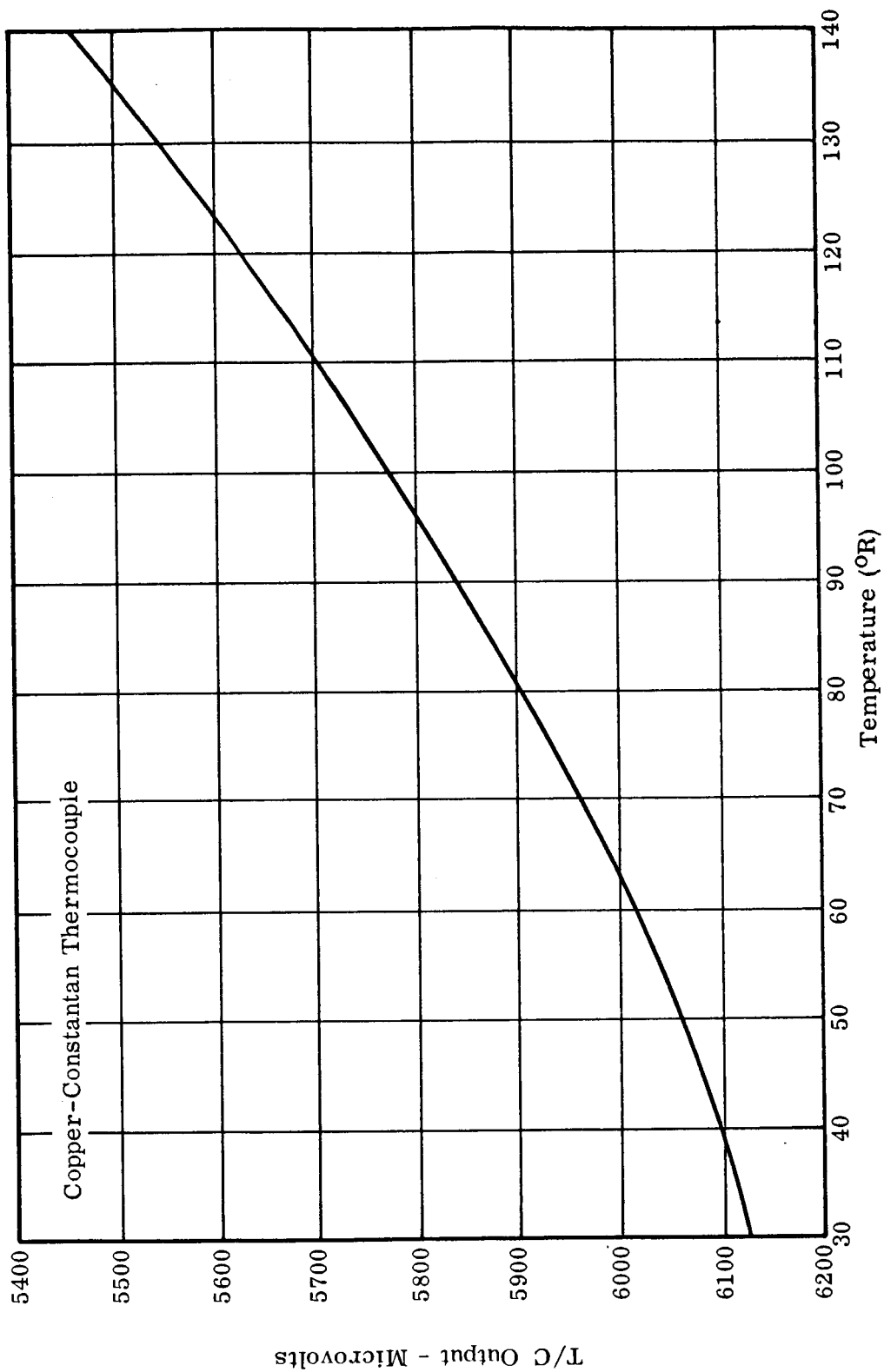


FIGURE 2 304 SST INSTRUMENTED TEST SPECIMEN, PLATINUM RESISTANCE THERMOMETER
COMBINATION PACKAGE IN TEST LOOP #4 - THERMOCOUPLE NO. 2

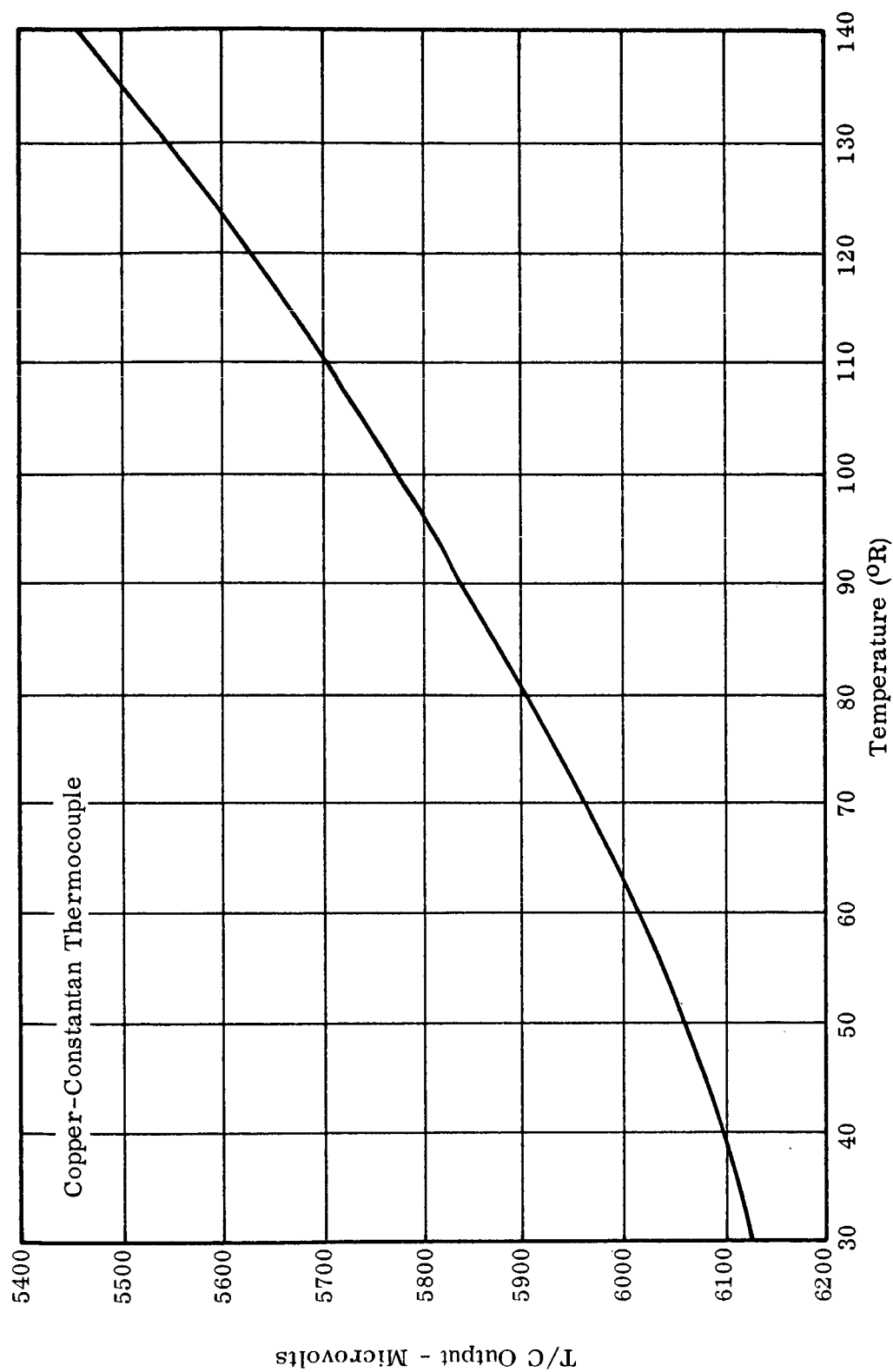


FIGURE 3 304 SST INSTRUMENTED TEST SPECIMEN, PLATINUM RESISTANCE THERMOMETER
COMBINATION PACKAGE IN TEST LOOP #4 - THERMOCOUPLE NO. 3

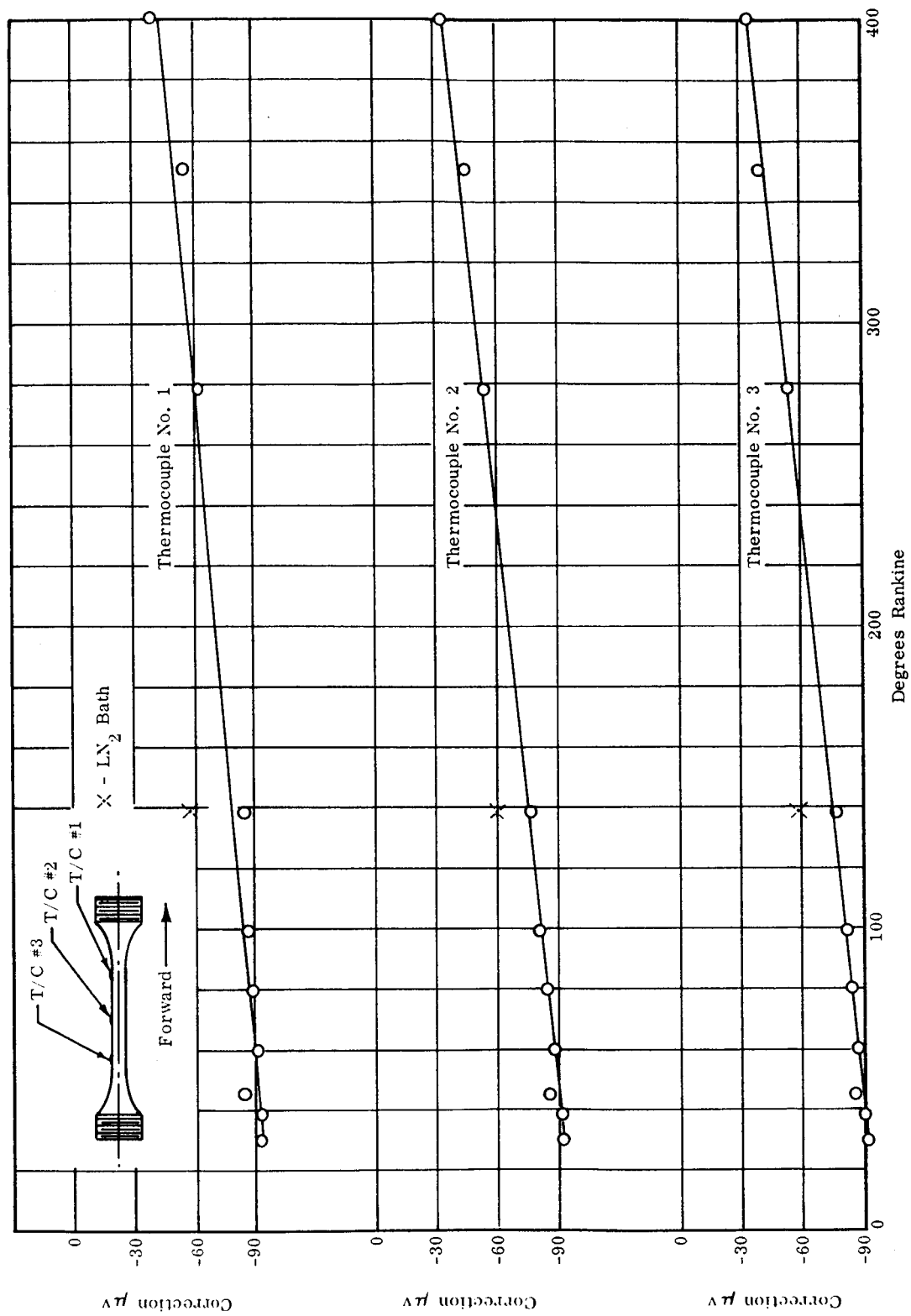


FIGURE 4 CORRECTION FACTORS FOR COPPER-CONSTANTAN THERMOCOUPLES FROM 300R to 400R

2.3 REMOTE HANDLING EQUIPMENT

The test loop transfer cask was probed for shielding uniformity by using an 11 curies cobalt 60 source. The results of the probe were satisfactory. This additional test of the shielding uniformity was required since, in the initial fabrication, the molten lead had not adhered to the stainless steel bulkheads in the cask. Upon cooling, shrinkage cavities were created adjacent to the bulkheads. These void areas were filled with pulverized lead as completely as possible, considering the cask configuration. As a measure to assure that these void areas were in fact filled, this check was made after the cask was received at the Plum Brook Reactor Facility. It was believed that in the event the pulverized lead had not filled the areas, the lead would have shifted during transportation with resultant radiation leakage. The check verified that the shielding uniformity of the cask is adequate. The junction between the upper and lower halves of the cask, however, is not uniform due to distortion of the stainless steel plate on the mating surfaces during final assembly operations. This distortion allows some radiation streaming, although it is not considered of a serious nature. The joint, however, will be made more uniform by removing the high spots on both the upper and lower halves to improve the mating surfaces of the cask.

The power supply system which is used with the remotely operated test loop transfer tongs has been installed by NASA in the hot cell area. The tongs were tested successfully using the dummy test loop. These tongs will be used in the hot cell area to remove the test loop from the test loop transfer cask and onto the support mounted on the hot cell door by which the test loop is moved into the hot cell. Operation of the tongs will take place from the viewing window outside the hot cell laboratory.

2.4 SAMPLE CHANGE SYSTEM

During this reporting period, one test loop carriage was modified as outlined in Progress Report No. 10 on pages 5 and 6. After the completion of preliminary tests, the carriage was used for the

flux mapping operation and operated very satisfactorily. A total in excess of twenty (20) insertions into the beam port was made with this carriage with no incident. Following satisfactory operation of this carriage, the other test loop carriages were modified similarly.

During the first attempt to insert Carriage No. 2 into HB-2 after modification, three of the cage axles failed when the carriage was approximately half way between the "normal rear" and "full forward" positions. The failures were of an inter-crystalline nature and occurred at the point of contact between the axle and the edge of the insert in the bracket. Two additional modified carriages had similar failures on the initial insertion into HB-2 and a fourth set developed one failure after approximately ten inches of travel against a constant force of 4350 lb applied with the fixture illustrated in Figure 5.

An investigation of the cause of failure was undertaken which consisted of physical testing of the remaining cages and metallographic examination of both satisfactory and failed cages. The physical testing was performed in the test figure illustrated in Figure 6. A ten ton hydraulic ram applied a load to the bearing surfaces of the rollers in a manner similar to the service load application. The force was measured with a gage which had been calibrated against the Hot Lab Instron testing machine immediately after the calibration of the Instron with dead weights. A cage from the initial set installed in Carriage No. 1 withstood a load in excess of 10,000 lb without failure while two from the lot in which failures were encountered failed at 3200 lb and 6200 lb. Hardness tests in both groups of cages were uniformly in the R_C 51-53 range.

Metallographic examination revealed that the cages which did not fail had a martensitic structure with uniform carbide dispersion throughout the acicular matrix normal for properly heat treated Type 420 stainless steel. This structure is illustrated in Figure 7.

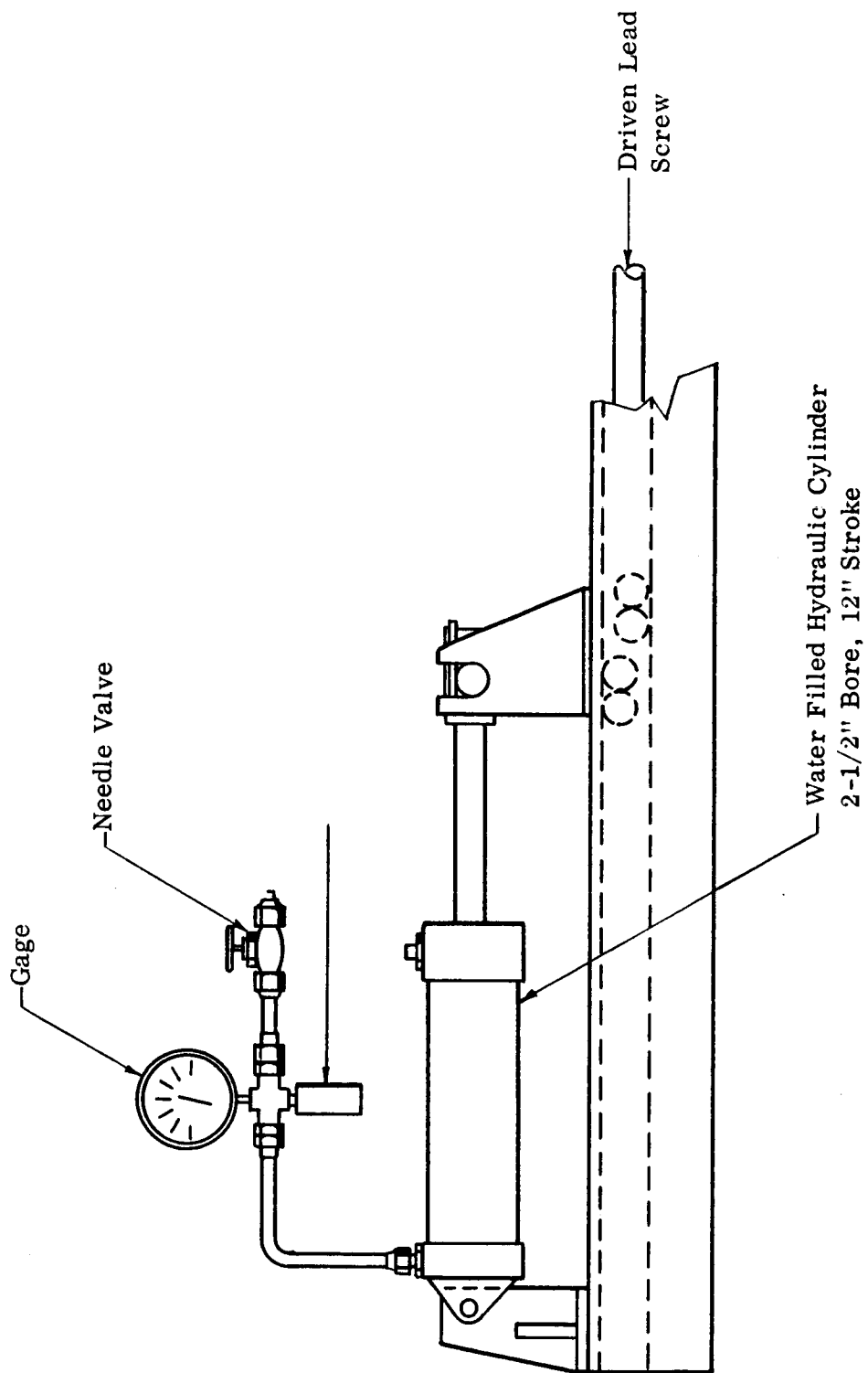


FIGURE 5 TEST FIXTURE FOR CHECKOUT OF TEST LOOP DRIVE SYSTEM

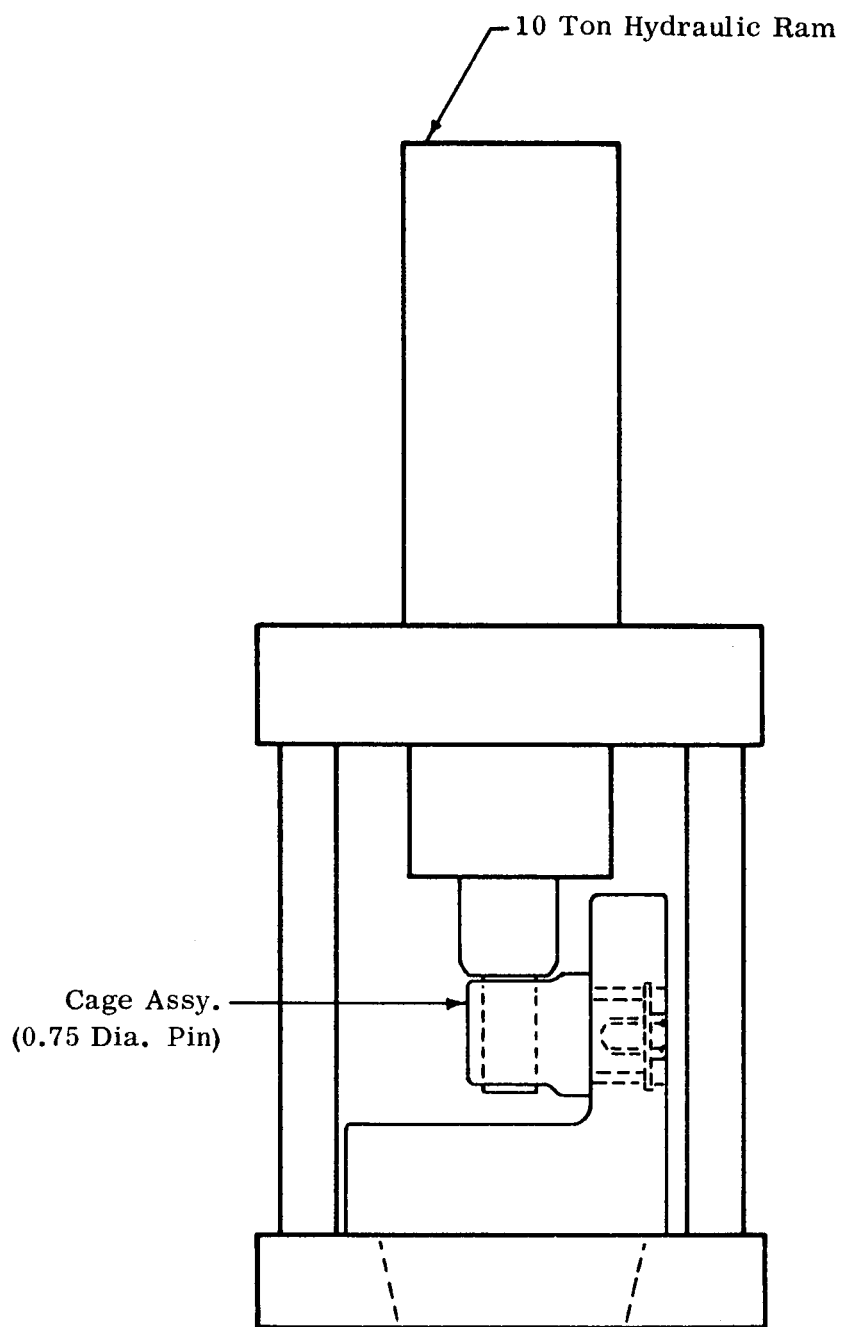


FIGURE 6 BEARING CAGE TEST FIXTURE

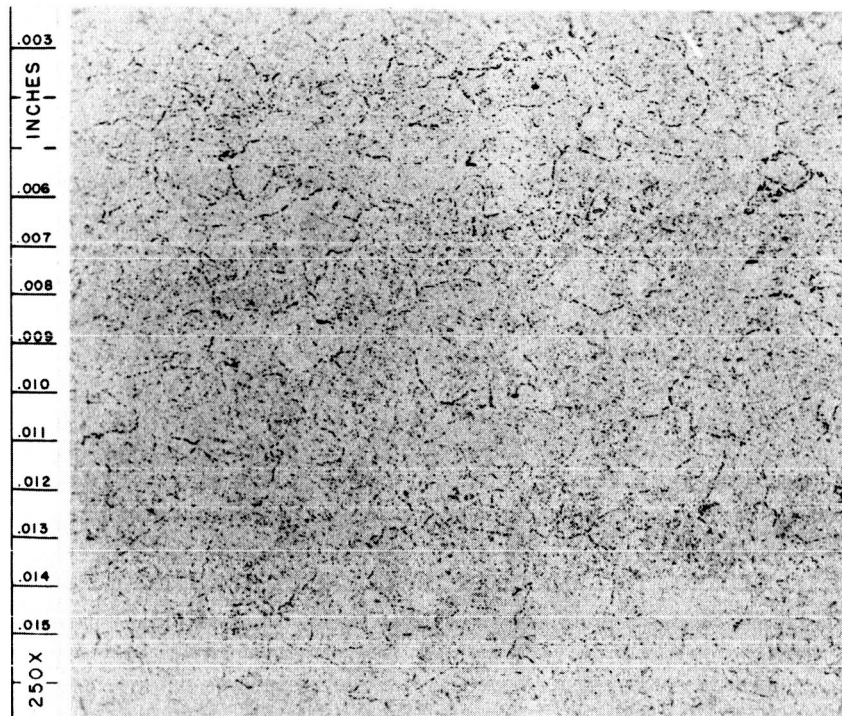


FIGURE 7
MICROSTRUCTURE
UNFAILED CAGE
X250

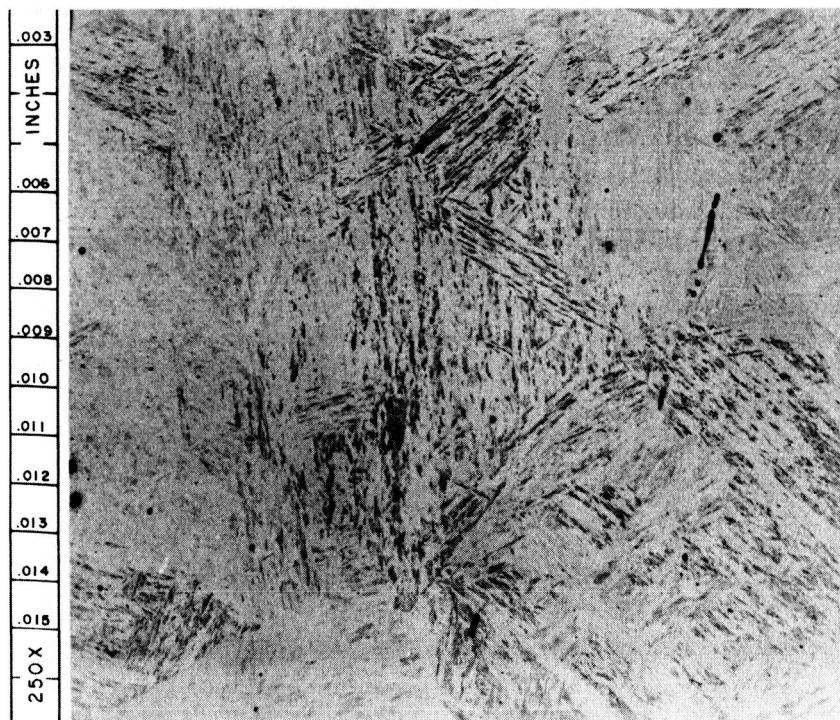


FIGURE 8
MICROSTRUCTURE
FAILED CAGE
X250

The failed carriage had a coarse Widmannstaetten structure, a brittle constituent associated with moderate overheating, containing undissolved carbides indicative of inadequate holding time at austenizing temperature. There also were intermettalic precipitates in the grain boundary area. This structure is illustrated in Figure 8.

The presence of undissolved carbides and intermettals at the grain boundary both would reduce the ductility and increase the notch sensitivity of the material. Type 420 stainless steel, even when properly heat treated, has rather low ductility and is somewhat notch sensitive. Any anomalies in the heat treating process can be expected to be reflected in an abnormal failure rate. Although the failure is attributable to an undesirable metallurgical structure resulting from heat treating practice, the specified hardness range was met and the quality of the heat treatment is probably average for commercial job-shop heat treating. In view of the difficulty of providing adequate inspection to insure an optimum structural condition, it was decided to make replacement cages of a material less sensitive to minor heat treating variations.

The cage design was modified (Drawing 108-4154-3) to increase the cross-section in critical areas, principally increasing the thickness of the web portion and increasing the diameter of the axle from 3/4" to 0.9", machining a radius at the neck of the axle to avoid stress concentrations at this point, and increasing the length of the axle by 1/8" to reduce stresses in this piece. Stress calculations on the new design show a maximum bending moment of some 90,000 psi.

Three test cages were made from Inconel X-750, heat treated using the "interrupted cooling after aging" technique to develop maximum mechanical properties, a minimum tensile strength of 170,000 psi and a minimum yield strength of 115,000 psi. These cages were proof tested using the fixture illustrated in Figure 9 in the Hot Lab Instron testing machine. The cages withstood loads of 10,000 lb without plastic behavior, with bending deflections of approximately 0.025" under load and complete recovery as

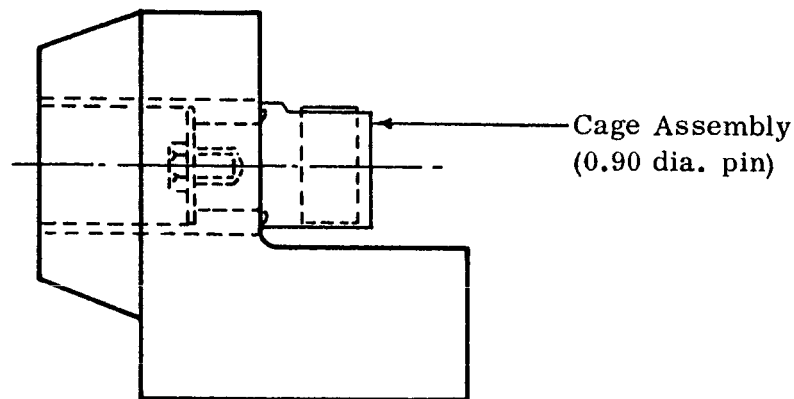


FIGURE 9 BEARING CAGE TEST BLOCK (INSTRON)

measured with the strain recording equipment of the Instron. The cages were not tested to failure because of the load limit of the Instron machine. However, a 10,000 lb proof load is considered sufficient assurance of satisfactory mechanical properties of the material and adequacy of the design since the design load is only 4160 lb. After testing, the cages were inspected using Zyglo penetrant (ZL-22) and no cracks or other indications were noted.

One further modification, imposed by the change in cage design, is changing the material of the brackets which support the cages from Type 304 stainless steel to Inconel X-750, and increasing the thickness of the bracket by 1/8" to allow for the increased length of the axle. This change to a high strength material is required because the increased diameter of the axle necessitated enlarging the mating hole in the bracket to a point where the section of the remaining metal is insufficient to provide an adequate margin of safety using allowable stress values for Type 304 stainless steel. These modifications were incorporated on appropriate design drawings.

One test loop carriage has been modified to this configuration and tested using a simulated load as supplied by means of hydraulic

cylinder relief valve. (Reference Figure 5.) Twenty (20) cycles were run on this fixture and a visual inspection following this phase of the testing program uncovered no defects in this system. The primary limitation of this test is that a movement of less than 1 foot can be obtained per cycle. Since the previous failures had occurred at the beginning of the cycle, it is reasonable to assume that a test of this nature will uncover any similar defects. A test loop has not been inserted into the beam port as of this date but approval to perform this task is expected shortly. No difficulty is anticipated in accomplishment of this insertion in view of the test results which have been accumulated thus far. Fabrication of the various components for the remaining carriages is also awaiting results of the insertion of the test loop in HB-2 against reactor primary coolant pressure.

The drive shaft of the Clevite hydraulic motor mounted on Carriage No. 2 was also broken in the failure which occurred during the insertion of a test loop. This drive shaft was replaced by the manufacturer and the motor returned to the Plum Brook Reactor Facility.

The hydraulic pump, manufactured by the Clevite Corporation in Cleveland, Ohio, was returned for replacement of seals due to water leakage past the seals and into the crankcase forcing the oil out the vent lines. This problem is still being studied by Clevite and the solution is expected shortly.

2.5 REFRIGERATION SYSTEM

The one set of helium transfer lines which had developed helium leaks through the inlet and outlet valves was sent back to the Vacuum Barrier Corporation for repair. The valve chest assembly in which these valves is located is of all welded construction so that the chest assembly had to be cut apart before access to the valves could be achieved. It was then necessary to cut the valves out of the system in order to determine the extent and cause of the damage. The valves are of the plug type and the plug showed some evidence of uneven and irregular seating

surfaces. The seats were also rather irregular, thus providing a good reason for the leakage. The cause for this malfunction has not yet been determined, although it seems feasible that some particulate matter has gone through both valves. Repair of these lines has been authorized and delivery is expected in late November or early December.

Helium leaks were also of concern in the other two sets of transfer lines. These leaks were on external joints and "on-site" repair was accomplished.

The refrigeration system, for the remaining portion of this report period, was used for training and in maintaining the proper temperatures of the flux measuring foils in the foil holder during the flux measurement operation in HB-2. It was also used in the temperature correlation tests described in paragraph 2.2.

2.6 TEST SPECIMEN FABRICATION

All test specimens expected to be required for the screening program as currently authorized have now been completed and are stored at the Plum Brook Reactor Facility. These specimens will be used for either in-pile or out-of-pile testing, depending on the scope of the testing program remaining to be conducted.

The majority of the tests remaining to be performed are in-pile tests but some of these specimens will be tested out-of-pile to complete the out-of-pile screening program, and in a few cases, to re-check data previously obtained.

3 FLUX MAPPING

Neutron flux and spectral measurements were made at various power levels with a 1/2" thick Mallory 1000 alloy shield plug and with a 1/2" thick Type 6061-T6 aluminum alloy shield plug installed in the beam port. Sets of foils consisting of neptunium-237, thorium-232, sulfur, nickel, magnesium and aluminum were used to measure the fast neutron flux in each irradiation. The nominal weights of the foils used in the flux measurements at these various power levels are shown in the table below:

Type Foil	Nominal Weight at 80 KW Run* with 1/2" Mallory 1000 Shield Plug	Nominal Weight at 39.93 MW Run with 1/2" Aluminum Alloy Shield Plug	Nominal Weight at 50.6 MW Run with 1/2" Aluminum Alloy Shield Plug
Np ²³⁷	18.0 micrograms	20.0 micrograms	20.0 micrograms
Th ²³²	4.0 grams	50.0 micrograms	50.0 micrograms
S	1.2 grams	50.0 micrograms	50.0 micrograms
Ni	1.0 grams	0.1 gram	0.1 gram
Mg	2.2 grams	0.5 gram	0.5 gram
Al	1.3 grams	0.5 gram	0.5 gram

All foils were held in-pile for 30 minutes during each irradiation and were counted and evaluated in accordance with Lockheed standard techniques. Results of these measurements are presented in Figure 10.

There is good agreement among results obtained in the four sets of measurements. It was expected that an increase of approximately 15% in fast neutron flux would result when the aluminum plug was installed in place of the Mallory 1000. This increase was not seen; however, it is likely that the increase was hidden within the probable error of measurement results.

* 2 runs made at 80 KW.

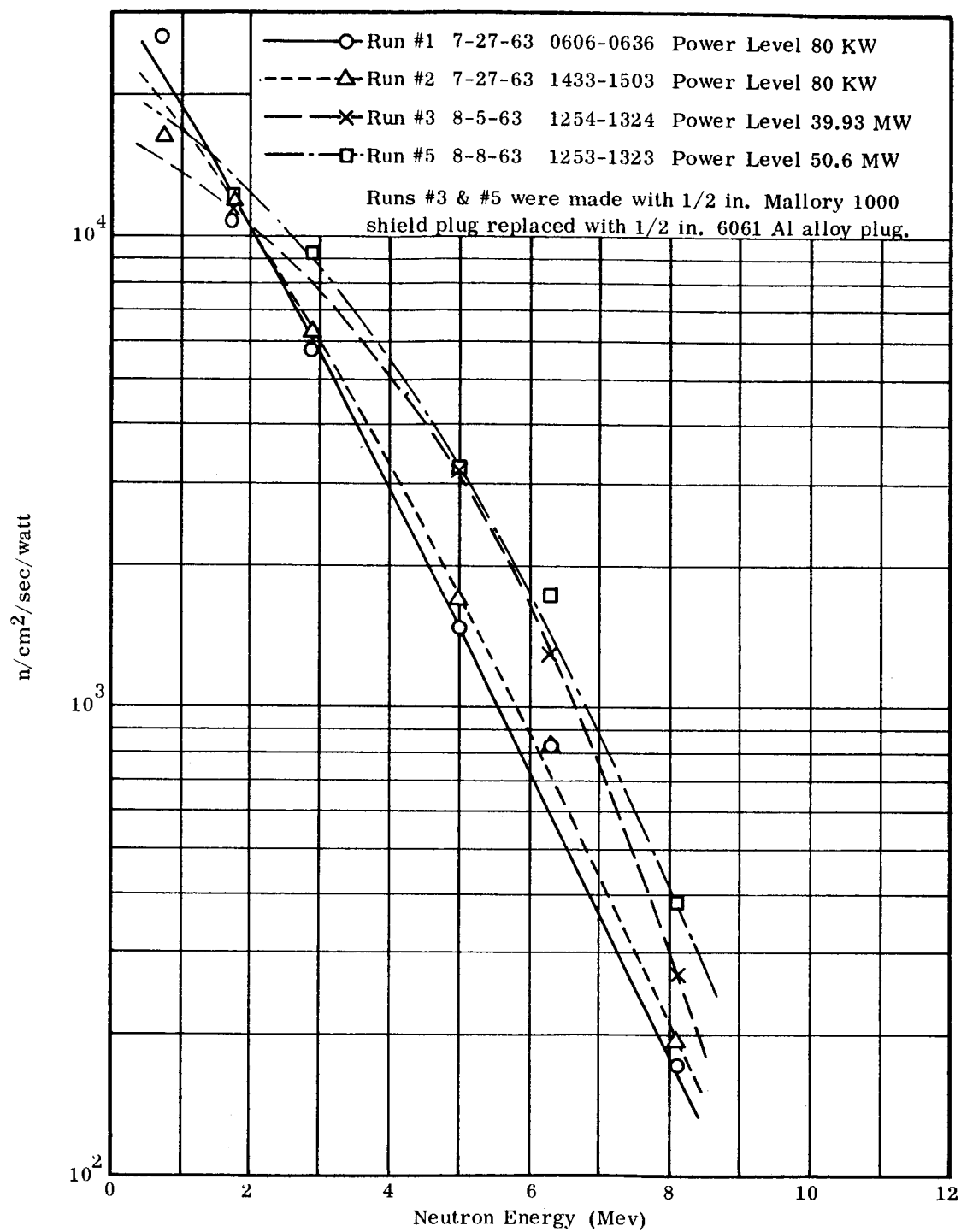


FIGURE 10 FAST NEUTRON FLUX MEASUREMENTS AT TEST SPECIMEN POSITION IN HB-2 BEAM PORT

Essentially no work was done with the fission counters and calorimeters with the cryostat inserted into the beam port with the Plum Brook Reactor at a high power level. It was observed that the calorimeter was giving an off-scale reading. This was due to the high gamma field encountered because of the reduced gamma attenuation resulting from the substitution of the aluminum alloy shield plug for the Mallory 1000 shield plug. The fission counter was also inoperative at the high power level run, as it was "swamped" with gammas.

Due to the fact that the fast neutron flux in HB-2 was found to be considerably lower than contemplated, an evaluation of these data was made to determine the proper shield plug to use to obtain the maximum possible neutron flux consistent with satisfactory operation of the nuclear instrumentation installed in the test loops. Evaluation, considerations and results were as follows:

1. The gamma dose rate in the test specimen location was found to be 2.8×10^9 r/hr (7.0 watts/gm), due to core gammas and beryllium target gammas.
2. From design calculations shown on pages 4-8-5, 4-8-6, and 4-8-7 of NM-111:
 - a. The gamma dose rate in test specimen located in the test loop head from core and beryllium target gammas with 3/4" Mallory shield plug is 1.08×10^7 r/hr (0.03 watts/gm).
 - b. The gamma dose rate in test specimen located in the test loop head captured by the tungsten in the shield plug is 3.2×10^7 r/hr (0.09 watts/gm).
 - c. The gamma dose rate at 1.65" depth with 3/4" Mallory 1000 shield plug in place is 0.12 watts/gm, which is the sum of a. and b. above.
3. The gamma dose rate, as measured by NASA personnel, was 2.4 watts/gm with an aluminum thimble and 0.02 watts/gm with the 3/4" Mallory 1000 shield plug installed.

4. The energy absorption mass attenuation coefficients used for the 0-1 Mev, 1-4 Mev and 4 Mev energy groups were 0.2, 0.03 and $0.04 \text{ cm}^2/\text{gm}$ respectively.
5. Maximum gamma dose rate on the nuclear instrumentation should be $5 \times 10^7 \text{ r/hr}$ (0.12 watts/gm).

The 0.02 watts/gm, as measured by NASA, was approximately a factor of 6 lower than design values calculated by LAC. The fast neutron flux as measured by LAC, reported in Section I, was a factor of 25 lower than expected. It is reasonable to assume that the incident thermal neutron flux was equally low. In this case, the gamma dose rate due to tungsten capture gammas should be reduced from 0.09 watts/gm to 0.004 watts/gm which, when added to the 0.03 watts/gm from core and beryllium target gammas, gives a value close to the 0.02 watts/gm measured by NASA.

By using the fast neutron removal cross section for tungsten, it was estimated that the fast neutron flux would be 10% less with the 3/4" plug than with the 1/2" plug.

Estimates based on a thermal neutron measurement made by LAC with a 1/2" aluminum plug show that the thermal neutron fluxes should be as follows:

- 1/2" Mallory Plug - $3 \times 10^3 \text{ n/cm}^2/\text{sec/watt}$
- 3/4" Mallory Plug - $1.3 \times 10^3 \text{ n/cm}^2/\text{sec/watt}$
- 1" Mallory Plug - $7 \times 10^2 \text{ n/cm}^2/\text{sec/watt}$

A measurement made by NASA at 5" at 60 mw with an aluminum thimble with rods "in" showed that the thermal neutron flux was $8.6 \times 10^{12} \text{ n/cm}^2/\text{sec}$. Attenuation calculations based on this measurement with the permanent Mallory 1000 shield and with the 3 plugs gave the following results:

- 1/2" Mallory 1000 Plug - $2.2 \times 10^3 \text{ n/cm}^2/\text{sec/watt}$
- 3/4" Mallory 1000 Plug - $1.1 \times 10^3 \text{ n/cm}^2/\text{sec/watt}$
- 1" Mallory 1000 Plug - $4.7 \times 10^2 \text{ n/cm}^2/\text{sec/watt}$

Further calculations show that with the above thermal flux with the 3/4" plug and with the measured fast neutron flux, the relative fission counter response is as follows:

$$\frac{\text{Thermal Fissions}}{\text{Fast Fissions}} = 4.5 \times 10^{-14} \times \frac{(\phi_{\text{th}})^2}{\phi_{\text{f}}}$$
$$= 1.6 \times 10^{-4}$$

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